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RESEARCH ARTICLE

INVESTIGATION OF THE POTENTIAL OF WASTE BONES AS A CATALYST IN BIOFUEL PRODUCTION

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ABSTRACT

It has recently come to light that inappropriate waste management (particularly of animal bones) is a major contributor to environmental degradation on a global scale. Instead, it would be extremely beneficial to the economy if the bioeconomy civilization could find novel applications for these discards. Biodiesel's output can be put to good use as a heterogeneous catalyst in the effective recycling of animal bone. Ca-based catalysts produced from discarded bones, have been extensively studied in the transesterification reaction due to their high catalytic activity and abundant feedstock. Calcium oxide, a common mineral, can be used to create heterogeneous catalysts (in the form of discarded bones). However, process improvements are required to make calcium oxide-based catalysts suitable for industrial use. This research summarizes the sources of waste animal bones, provides context for the recent advances in the development of various Ca-based catalysts derived from waste animal bones, characterized a calcium oxide-based catalyst derived from waste animal bones, and details its application in biodiesel production. In this work, waste bones were modified by calcination at 950^{0}C for 3h. The catalyst was analyzed using scanning electron microscopy (SEM) for its morphological structure, Fourier transform infrared spectroscopy (FTIR), for its functional group, X-ray fluorescence (XRF), for its elemental composition, thermogravimetric analysis (TGA), for thermal stability and Brunauer Emmett Teller (BET) surface area analysis. Overall, raw sample was found to have no effect on the waste bone, while high temperature calcination greatly affected the pore size, surface area, composition, and thermal decomposition profile of the waste bone sample. According to the study's findings, used animal bones can be used to create catalysts that are appropriate for trans-esterifying a variety of oil sources into high-quality biodiesel. This increases the total energy and atom-efficiency of both conventional and new chemical processes while also making the system more cost-effective and ecologically benign. This discovery will encourage scientists to look into possible wastes of bones and pave the way for a more cost-effective and ecologically friendly way to make biodiesel and should also continue to be evaluated for the likelihood of their use in the commercial sector.

KEYWORDS

Characterization, Waste bone, Calcium oxide, Calcination, Heterogeneous catalyst

1. INTRODUCTION

Due to environmental concerns resulting from the continuous reliance on fossil fuel sources, current industrial energy demands, and an uptick in the global population, the use of solid-based catalysts in the field of renewable energy to produce renewable fuel has become a significant area of research (Adeyinka et al., 2018). To prevent environmental anomalies that could become severe if the discovery of different energy sources is disregarded, it has become important and demanding to replace fossil fuels with similar renewable fuels (Soudagar et al., 2021). In the last few decades, there has been a worldwide trend toward using biofuels. Biodiesel is often seen as a viable replacement for mineral diesel in many nations due to its reputation as a renewable, sustainable, and non-toxic biofuel (Khan et al., 2019; Mujtaba et al., 2020). For many compression ignition engines, biodiesel is an attractive alternative fuel due to its minimal environmental impact, great recycling potential, and low transportation costs (Omojola et al., 2020). But biodiesel doesn't have to be seen as a direct replacement for petroleum diesel. Instead, it can be seen as a part of a reliable and diverse energy source (Mazaheri et al., 2021).

An appropriate catalyst is required for the transesterification process (Walid et al., 2022). In the presence of acid or base catalysts, vegetable oil or animal fats are mixed with alcohol, usually ethanol and methanol, to make Fatty Acid Methyl Ester (FAME) or biodiesel (Verma and Sharma, 2016; Folayan et al., 2019). Based on their physicochemical characteristics, homogeneous, heterogeneous, or enzymatic catalysts can be assigned to both acid and base catalysts (Amini et al., 2017; Thangarasu and Anand, 2019; Ong et al., 2021). Properly supported catalysts should be created to improve catalyst performance, prevent unneeded leaching of active species, and prevent reaction byproducts from clogging the catalyst. Heterogeneous catalysts can be used more than once, which could increase the amount of biodiesel that can be made (Walid et al., 2022). Due to the drawbacks of homogeneous catalyst use, it was vital to develop and use heterogeneous catalysts. The focus on CaO (alkaline earth oxides) oriented heterogeneous catalysts has increased as a result of the solubility

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and dissolving tendencies of other media (Ameh et al., 2021). As a result, current research has focused on a variety of heterogeneous base catalyst categories, including metal oxides, calcined hydrotalcite, supported alkali metals, and anion exchange resins (Hussain et al., 2021).

Magnesium oxide (MgO), barium oxide (BaO), and calcium oxide (CaO) are the most commonly used heterogeneous alkali catalysts. The cost of production, which includes costs for the catalyst, purification, and feedstock, is one issue that prevents biodiesel from being competitive and from being produced on a large scale (Tamrat et al.,2022). Calcium oxide (CaO) stands out among base heterogeneous catalysts because it exhibits qualities including strong basicity, mild reaction conditions, low cost, increased biodiesel output, and least solubility in fuel (Hussain et al., 2021). Recently, a lot of researchers have redirected their attention to designing and developing catalytic materials from affordable and wasteful renewable sources to increase the process's overall sustainability (Bennett et al., 2016). There are several calcium-containing waste products available worldwide (Hussain et al., 2021). Therefore, one option to lower production costs is to use different types of catalysts made from waste materials. Animal waste bones have the potential to be used as a source of heterogeneous catalyst, which could lower the cost of biodiesel manufacturing. Most of the solid waste that comes from slaughterhouses is animal bones, which make up 30.4% of all cow waste (Tolera et al., 2020).

Catalysts derived from locally sourced resources, such as calcined fish scale, calcined scrap bone, and mollusk shells, have been used to lower production costs for biofuels without sacrificing efficiency (Oladipo et al., 2018; Marwaha et al., 2018). Waste bone can become poisonous if not handled and disposed of properly despite the fact that it includes a number of bioactive materials and other precious minerals with known commercial importance (Zaman et al., 2018). Abattoir waste bones damage the environment by encouraging the growth of pathogenic germs by providing oxygen (Tamrat et al., 2022). The most efficient way to manage waste is to turn it into useful and valuable materials using environmentally friendly technologies (Hussain et al., 2021). Animal waste bones that pollute the land can be effectively converted into useful materials like catalysts. Waste bone can be improved through processing and alteration to boost its worth and usefulness. According to research, bones that were previously thought of as trash can be recovered, repurposed, and renewed for use (Chung et al., 2019; Hamzah et al., 2020).

Using animal bone as a catalyst in the creation of biofuels has various benefits (Etim et al., 2020). The catalyst can be distributed widely and effectively because of the high porosity and large surface area of hydroxyapatite [Ca10 $(PO4)_6$ $(OH)_2$], which is full of calcium and phosphorus inorganic minerals with good thermal stability and porous crystal structure that aids transesterification reaction through the surface-active site present in bones (Ramesh et al., 2018; Gebeyehu et al., 2020).

Several scientists have conducted biofuel synthesis studies using waste bones as an eco-friendly catalyst, and the findings are encouraging. Using 60 OC reaction temperature, a 15:1 methanol to oil molar ratio, a 4-hour reaction period, and a 5-weight % catalyst loading, ostrich bone that had been calcined at 900^{0}C was also used to produce 90.56 weight % of FAME from spent cooking oil (Khan et al., 2020). That produced biodiesel with a concentration of 96.78% from palm oil using calcined waste animal bone as a catalyst at 65^{0}C, 200 rpm, and a catalyst to oil to methanol ratio of 1:18 (Obadiah et al., 2012). reported a 97wt% yield of soybean oil base FAME produced utilizing calcined animal bone with reaction conditions of 65^{0}C, 3h, and a 6:1 methanol to oil ratio (Smith et al., 2013). Furthermore, by calcining waste animal bone at 9000C with transesterification reaction parameters of 70^{0}C reaction temperature, 3h reaction time, and a 9:1 methanol to oil molar ratio, 96.1wt% yield of FAME was obtained from the bone (Nisar et al., 2017). More than 95% yield for canola oil-based biodiesel was shown when they examined a sheep bone-derived catalyst at the calcination temperature of 600^{0}C (calcined for 8 h) by (Ghanei et al., 2016). Similar to this, in another location, ultrasonic assisted transesterification with a catalyst derived from chicken bone produced more than 95% biodiesel in 32.08 minutes (reaction time) (Ansari et al., 2020). In a different study, a novel layered heterogeneous catalyst made from waste animal bones was created in a two-step process that involved calcination and a hydrothermal reaction to increase biodiesel yields (Chingakham et al., 2019). In order to achieve 92% yield, used waste goat bone as a catalyst that was calcined at 900 ^{0}C for 3 hours with algae oil at a methanol to oil ratio of 11:1, catalyst weight of 2wt%, time: 180 min, at a temperature of 60^{0}C (Mamo et al., 2019).

As there has been recent discussion of using waste bone as a catalyst for a transesterification reaction, the question of how well waste bone has been changed and characterized has arisen as the driving force behind the present study. This question serves as the inspiration for the current investigation. Calcination is one of the methods used to modify the waste bone. By enhancing important catalytic fingerprints and other performance indicators including surface area, pore volume, molecular and crystalline structure, calcination promotes the synthesis and modification of catalysts (Omojola et al., 2020). In order to better understand how waste bones, behave when subjected to various characterization techniques, the current study modified and characterized waste bones. Calcium oxide (CaO), which is obtained from waste bone, was created in the current attempt from powdered raw and calcined waste bones and then was submitted to characterization. Analysis, comparison, and discussion of the characterization's result were conducted.

2. Materials and Methods

2.1 Materials/ Method

The used trash bone came from a neighborhood market in Abuja, Nigeria. To get rid of anything stuck to the waste bones' bodies, they were immersed in hot water for seven hours. To remove contaminants, it was rinsed with tap water first, and then with deionized water. To remove moisture, the bones from the garbage were dried in an oven kept at 110^{0}C for 4 hours. Using a mortar and pestle and a laboratory grinder, the dried sample was manually crushed and mechanically ground. The powder was run through a sieve with a mesh size of 100 m. The sample was then subjected to a 3-hour calcination process in a furnace with a temperature of 950^{0}C in order to completely convert the waste bone $CaCO_3$ to CaO with 10°C/min heating rate. The calcined sample was removed from the furnace after cooling to room temperature. A covered plastic jar was used to store the leftover bone powder. X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA)/Derivative thermogravimetric (DTG), scanning electron microscope (SEM), and Brunauer-Emmett-Teller were used to characterize the raw and calcined samples (BET). The flow diagram of catalyst preparation is shown in figure 1.

2.2 Characterization

The Brunauer-Emmett-Teller (BET) method, based on the nitrogen adsorption/desorption principle and acquired at 77 K and 60/60 s (ads/des) equilibrium period, was used to analyze the textural characteristics of the produced samples. The catalyst sample's exterior appearance, crystal structure, and orientation were all determined using a scanning electron microscope (SEM). The catalyst surface's functional groups were identified using Fourier transform infrared radiation (FTIR) spectroscopy. The catalyst samples' distinctive fingerprints and structural properties were verified using X-ray diffraction (XRD). At room temperature, diffraction patterns were produced using cuk radiation (1.54051), with scanning angles of 2 of 4°–90° and 8–24° for calcined and raw, respectively. Utilizing a Varian XRF analyzer, X-ray fluorescence (XRF) was used to ascertain the chemical make-up of the produced catalyst.

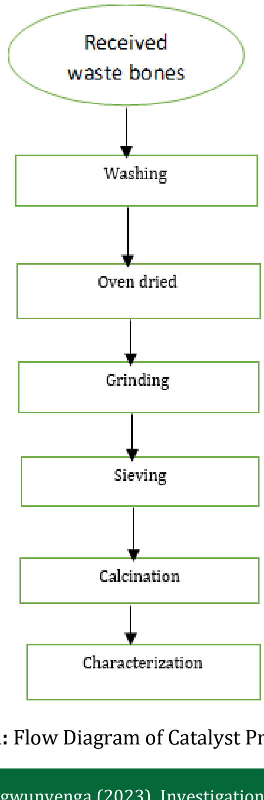

Figure 1: Flow Diagram of Catalyst Preparation

3. RESULTS AND DISCUSSIONS

3.1 SEM Analysis

The surface morphology of discarded bone powder was analyzed by scanning electron microscopy to see the impacts of raw and high-temperature calcination. The outcomes are depicted in (Figure 2 and Figure 3). Raw and calcined samples showed the same particle shape in SEM pictures. The particles in both samples were of various sizes and shapes. In Fig.2, we see a crumpled and rough surface covered in hair line cracks and tiny hole-like surfaces, all of which are designed to create a high density of pores that act as flow channels within the structure. But as can be shown in Fig.3, the calcined sample provided with more irregularly shaped particles. The calcination process had an effect on the sample, which explains the result. Because of their smaller dimensions, grains and aggregates may have greater specific surface areas. $CaCO_3$ was thermally degraded to CaO and CO2 throughout the calcination process (Onwubu, et al., 2017).

3.2 FTIR Analysis

The functional group and structure of the raw and calcined sample were investigated using FTIR. Figure. 4 and Figure 5, showed the results of FTIR analysis performed on raw and calcined waste bone powders, respectively. The raw sample showed prominent absorption bands at 3839 cm $^{-1}$, 2922 cm $^{-1}$, 2322 cm $^{-1}$, 1636 cm $^{-1}$, 1241 cm $^{-1}$, 1010 cm $^{-1}$, and 872 cm $^{-1}$. There are asymmetric stretch, out-of-plane bend, and in-plane bend vibration modes, and they are all responsible for this. Absorption peaks in the calcined sample were observed at 3626 cm $^{-1}$, 2918 cm $^{-1}$, 1617 cm $^{-1}$, and 950 cm $^{-1}$. Sharp stretching band at 3626 cm $^{-1}$ was observed in the calcined waste bone, which can be attributed to the presence of the OH^{-1} group (Joshi et al., 2015). The carbonate in the waste bone is converted to CaO during the calcination process, and the $CO^2{}_3$ molecules' absorption bands are shifted upwards as a result of the heat treatment. It is theorized that this change is due to the lighter functional group attached to the $CO^{-}{}^2{}_3$ ions.

3.3 XRD Analysis

The XRD profiles of raw and calcined waste bone samples are shown in (Figures 6 and 7), respectively. The profile of the raw sample with 2θ scan range of 4.0-90^{o} and the calcined sample presented a remarkably distinct displaying a broad peak at 220, 220, 240, and 240, respectively. CaCO3 was calcined to CaO and Ca $(OH)_2$ in the sample. As can be seen, the main components of calcined waste bone are calcium oxide and calcium hydroxide. Minimal peaks can be seen in the XRD profile, indicating the formation of Ca $(OH)_2$. Due to the high temperature calcination, $CaCO_3$ is transformed into CaO during the thermal treatment. There may be Ca $(OH)_2$ in the calcined sample because CaO reacted with air while being packaged and analyzed. Sharper peaks can be seen in the XRD pattern of the calcined waste bone compared to the raw sample, indicating improved crystallinity (Figure 7). Higher surface area, a key feature of a heterogeneous catalyst, was also observed, as was the particle size reduction of the calcined waste bone spectra.

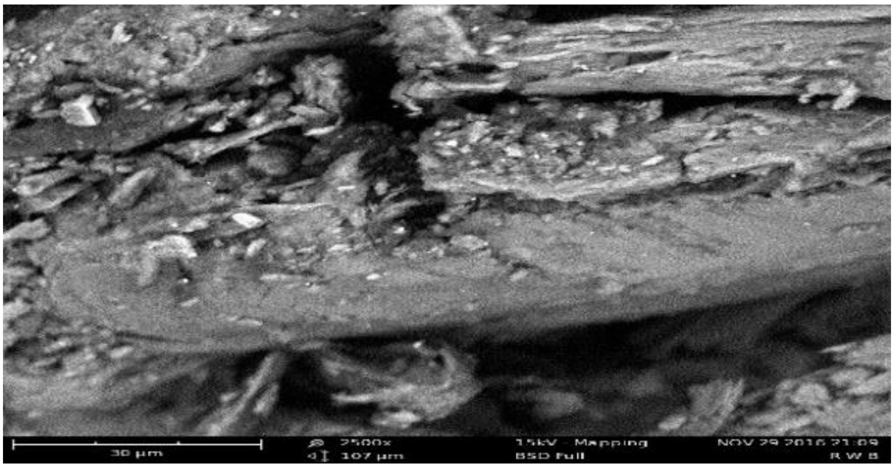

Figure 2: SEM image of raw waste bone

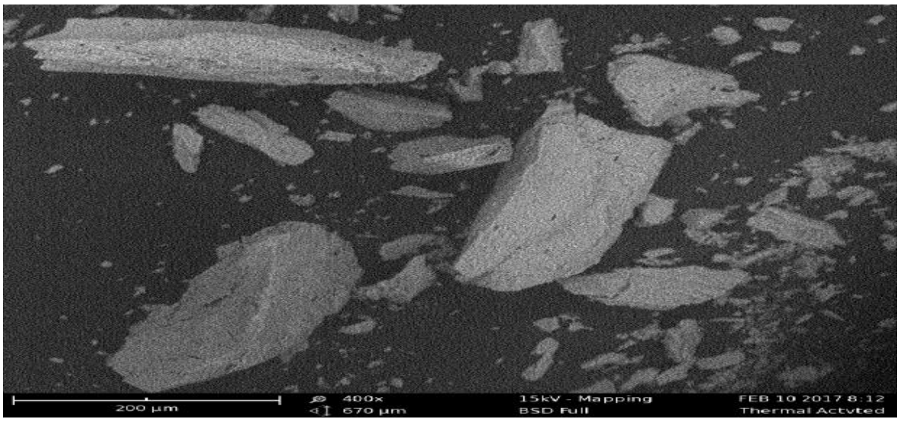

Figure 3: SEM image of calcined waste bone

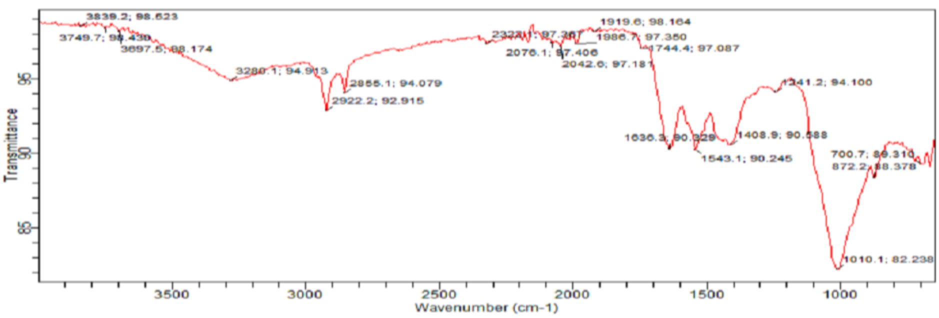

Figure 4: FTIR image of raw waste bone

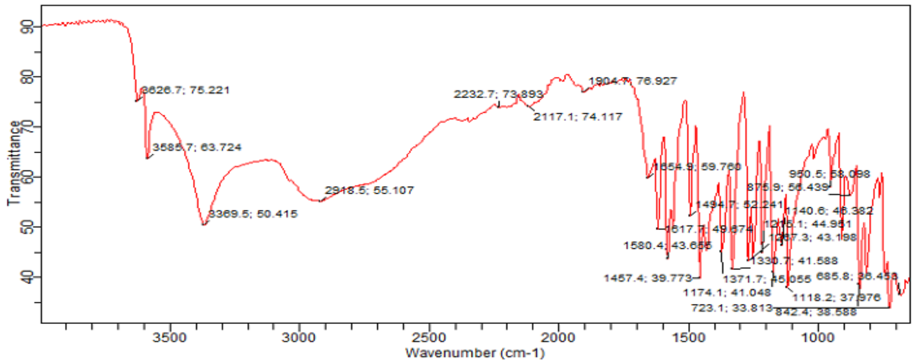

Figure 5: FTIR image of calcined waste bone

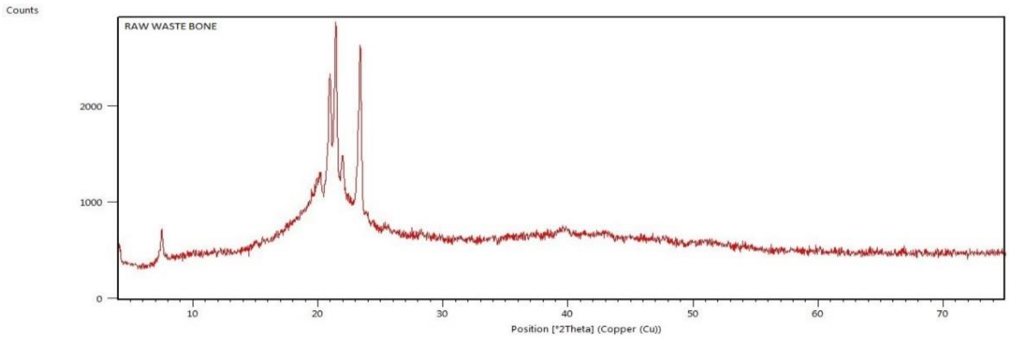

Figure 6: XRD profile of raw waste bone

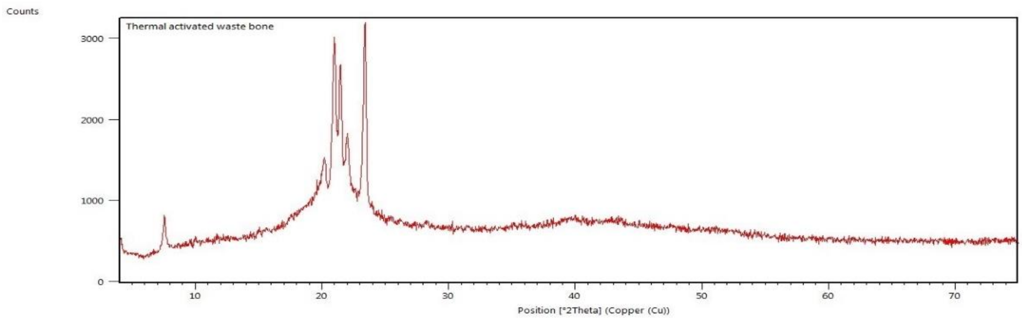

Figure 7: XRD profile of calcined sample

3.4 X-ray Fluorescence (XRF) Analysis

Analysis by X-ray fluorescence spectroscopy (XRF) of both raw and calcined waste bone revealed the presence of many elements. The most common elements are calcium and phosphorus. Raw catalyst is more calcium-rich than calcined waste bone. However, the calcined sample has a larger concentration of phosphorus. It makes sense that calcium phosphate and carbonate would be prevalent if there were plenty of calcium and phosphorus. According to Tables 1 and 2, the percentage concentration of chromium in raw and calcined samples is 0.000. CaO measured by the XRF analyzer implied that the raw sample contained a high proportion of $CaCO_3$, and the dominance of CaO in the calcined sample was ascribed to the release of CO_2 during the calcination process.

3.5 Thermogravimetric Analysis (TGA) /Differential Thermal Analysis (DTA)

Figures 8 and Figure 9 show thermogravimetric and differential thermal analysis (TGA/DTA) curves, which illustrate and compare the thermal behavior of the samples during thermal decomposition in a controlled environment. The TGA curves for the raw and calcined samples were very similar, indicating a single-stage decomposition. Figure 8 shows the results of a thermogravimetric analysis (TGA) performed on raw waste bone, which revealed a 2% and 20% loss in weight between 100^{0}C and 150^{0}C ,350^{0}C and 420^{0}C, respectively, as a result of the evaporation of adsorbed water and the decomposition of carbonate. Weight was not lost at all across the entire temperature range of 4500C–9500C, which is thought to be the result of gradual dihydroxylation. The endothermic peak in the DTA curve appears between 250 and 3000 C, most likely caused by the evaporation of all water. As can be seen in (Figure 9), the TGA results for the calcined sample show a loss of mass between 100^{0}C and 200^{0}C (2% loss) and 300^{0}C and 400^{0}C (4% loss) due to the dehydration of the precipitating complex, the loss of physically adsorbed water, and the decomposition of organic matters. Rising losses in mass suggest that carbon is being extracted alongside water and gases (Abdel et al., 1981). Carbonate decomposition may also contribute to low mass loss. When heated above 400^{0}C, weight loss slowed but did not stop. Phase stability due to complete decomposition and possibly liberation of bound liquids and gases was observed within this temperature range, indicating a nearly stable curve. The breakdown of organic matter and carbonate are both contributors to the endothermic peak between 250^{0}C and 300^{0}C.

3.6 Brunauer- Emmett- Teller (BET) Analysis

Surface area, total pore volume, and pore diameter were measured to characterize the granular texture of the raw and calcined samples, respectively. The Brunauer-Emmett-Teller (BET) model was used to calculate the sample surfaces. The Dubinin-Radushkevich (DR) model was employed to ascertain the pore volume and the mean pore diameter. Data are shown in Table 3. The results showed that the calcined sample had better textural properties after calcination, which can be attributed to the activation of previously inactive sites caused by the removal of adsorbed gases and organic matter. According to Table 3, the calcined sample has a higher BET surface area (97.34 m2/g) and a higher pore volume (0.110 cm3/g) than the raw sample (11.23 m2/g), both of which indicate that the calcined sample is dominated by active molecular sites and is thus likely to result in better and faster interaction between the catalyst and the feedstock (Kumar et al., 2012). It is evident that sintering at high temperatures increases the surface area, pore volume, and decreases the pore size of the calcined sample compared to the raw sample. In turn, this causes the pores to enlarge and the intercellular walls to break down as a result of dehydration, resulting in less porousness. Non-thermal treatment reduced the sample's surface area, pore volume, and pore diameter.

Table 1: Elemental Weight Compositions of Raw Waste Bones		
Elements	**Confidence level**	**Concentration percentage (wt %)**
Na_2O	100	0.724
MgO	100	0.496
Al_2O3	100	1.177
SiO_2	100	5.339
P_2O_5	100	26.451
SO_3	100	1.777
Cl	100	0.386
K_2O	100	0.341
CaO	100	61.076
TiO_2	100	0.470
Cr_2O_3	100	0.000
Mn_2O_3	100	0.005
Fe_2O_3	100	1.467
ZnO	100	0.588
SrO	100	0.125

Table 2: Elemental Weight Compositions of Calcined Waste Bones		
Element symbol(oxides)	**Confidence level**	**Concentration percentage**
Na_2O	100	0.717
MgO	100	0.790
Al_2O_3	100	0.550
SiO_2	100	2.371
P_2O_5	100	34.562
SO_3	100	0.173
Cl	100	0.132
K_2O	100	0.380
CaO	100	59.951
TiO_2	100	0.0083
Cr_2O_3	100	0.000
Mn_2O_3	100	0.002
Fe_2O_3	100	0.129
ZnO	100	0.031
SrO	100	0.129

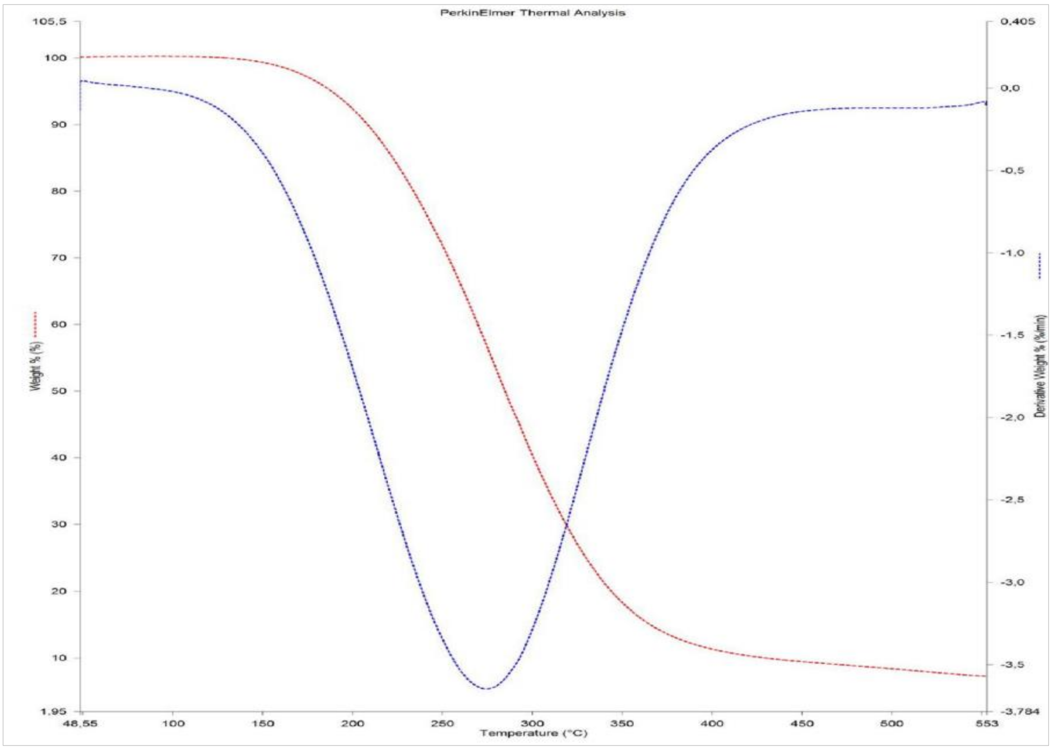

Figure 8: TGA/DTA thermogram of raw sample

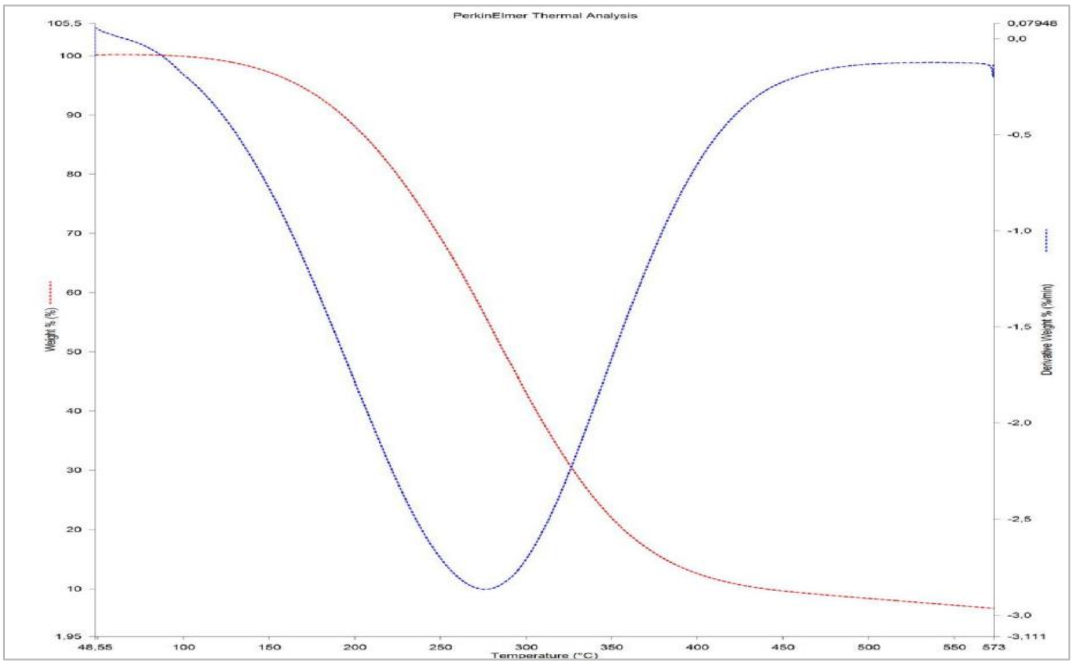

Figure 9: TGA/DTA thermogram of calcined sample

Table 3: Surface Area and Porosity Analysis of Raw and Calcined Sample

Sample name	Raw waste bone	Calcined sample
Surface area (m^2/g)	11.234	97.34
Pore volume (cm^3/g)	0.004	0.110
Pore size A°	5.901	1.672

4. Conclusion

The present research accomplished the development and characterization of a heterogeneous catalyst made from waste bone. The waste bone was found to be unaffected by the raw sample after characterization, but calcination at a higher temperature increased the surface area, pore size, and aided in the thermal degradation of CaCO3. As shown by BET analysis, calcination has a beneficial effect, particularly on the material's textural properties. Phase changes were observed by XRD analysis following calcination of the raw sample. The scanning electron micrograph of a calcined sample showed a number of pits and fissures in its surface. However, the characterization results suggest that the calcined sample at 950^{0}C may have higher catalytic activity than the raw sample thereby increased the surface area, pore size, and aided the thermal degradation of $CaCO_3$. By converting waste bone to catalyst, not only will the substantial reliance on synthetic commercial CaO for catalytic biofuel synthesis and other industrial utilizations be greatly reduced, but waste recycling and the proper disposal of the large quantity waste bones generated by abattoirs will be aided, minimizing the cost of waste disposal and reducing landfill waste. Calcination of waste bones at varying temperatures has the potential to be a useful source of CaO, but more research is needed to confirm this. As the world increasingly relies on renewable energy sources, more research is needed into the availability, source, and sustainability of waste bones catalyst to meet the demand of biofuel synthesis. In conclusion, all waste animal bones can be efficiently utilized as a heterogeneous catalyst for biodiesel production. Evaluation of the economic viability of biodiesel production using heterogeneous catalysts produced from discarded animal bones is urgently needed as this would be a renewable energy source with the added benefit of environmental protection. The widespread use of waste bone catalyst will also benefit the economy and produce promising catalysts from the calcium oxide waste materials. However, the catalysts that are produced are unstable and have issues with leaching during the reaction. A substantial challenge still exists in the production of efficient and catalytically stable calcium oxide catalysts. As a result, it is necessary to continuously try to change the technology in order to increase sustainable biodiesel production and lower the prices.

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